LLNL- PROC- 512881

Corrosion-resistant multilayer coatings for the 25-75 nm wavelength region

Regina Soufli

Lawrence Livermore National Laboratory

2011 International Workshop on EUV and Soft X-ray Sources, University College Dublin, Ireland
9 November, 2011

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Contributors

- ***Lawrence Livermore National Lab*:**

 Mónica Fernández-Perea, Jeff C. Robinson, Sherry L. Baker

 Jennifer Alameda

- ***Lawrence Berkeley National Lab*:** Eric M. Gullikson

- ***Evans Analytical Labs (Sunnyvale, California):***

 Harry Kawayoshi, Jiafang Lu, Martin Izquierdo

Regina Soufli
regina.soufli@llnl.gov

Overview

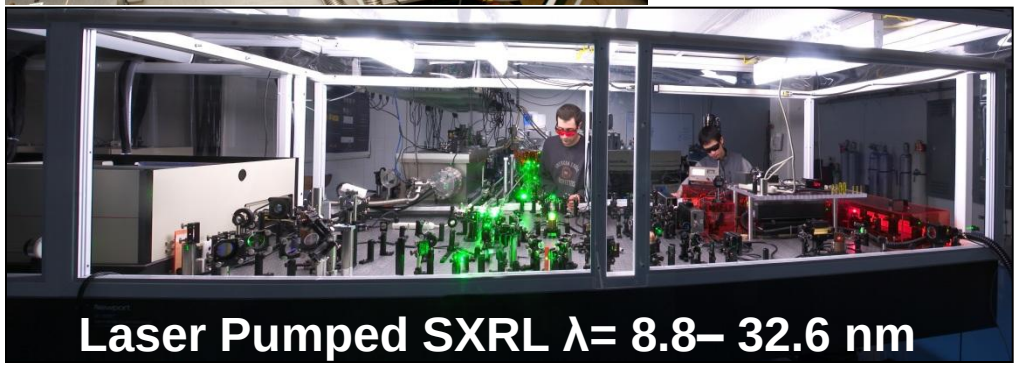
- Applications in the 25-75 nm region that could benefit from high-performance SiC/Mg multilayers
- Study of SiC/Mg corrosion: origins, mechanisms of propagation, impact
- Development of new, corrosion-resistant SiC/Mg multilayers
- Experimental reflectivity results
- Future multilayer designs

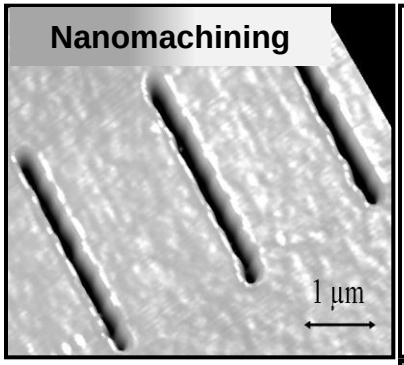
Regina Soufli
regina.soufli@llnl.gov

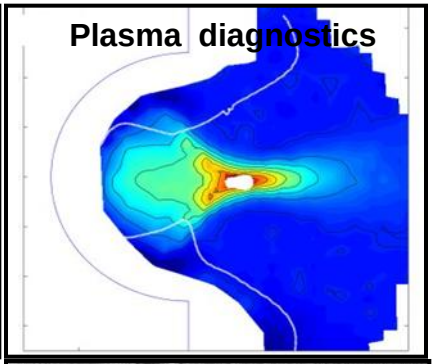
Compact plasma-based EUV/soft x-ray laser applications need multilayer coatings as collector and imaging elements

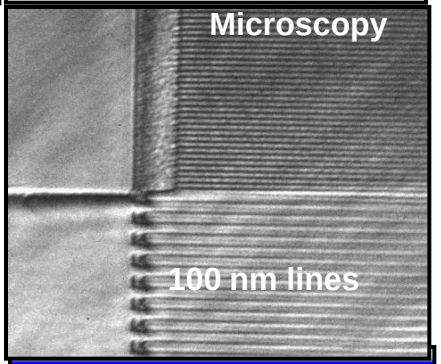


Courtesy: Jorge Rocca and Carmen Menoni, Colorado State University

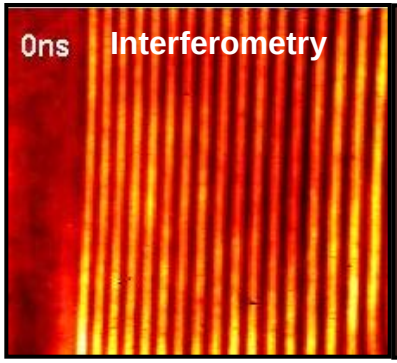


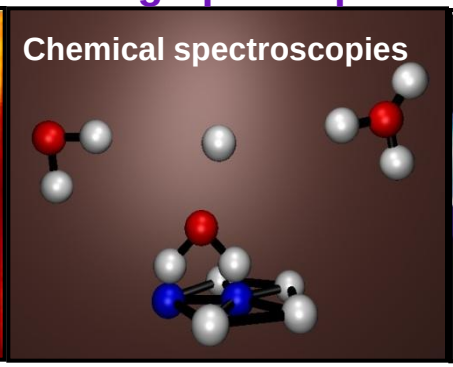


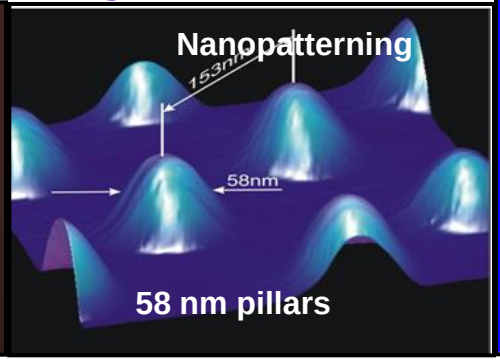


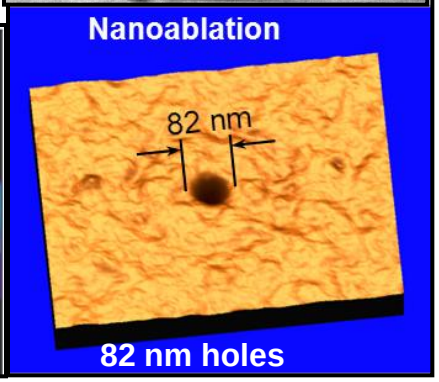


- High pulse energy (μJ-mJ)
- High monochromaticity ($\lambda/\Delta\lambda < 10^{-4}$)
- High peak spectral brightness

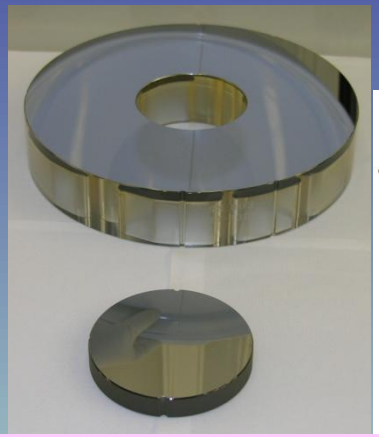




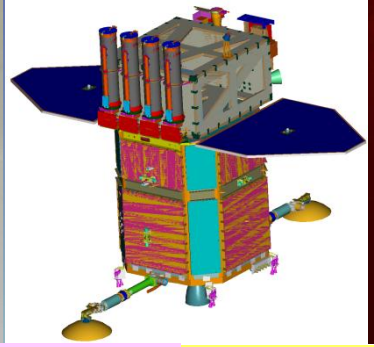


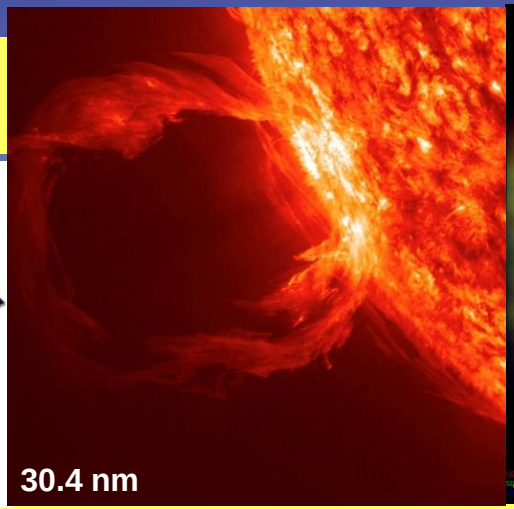


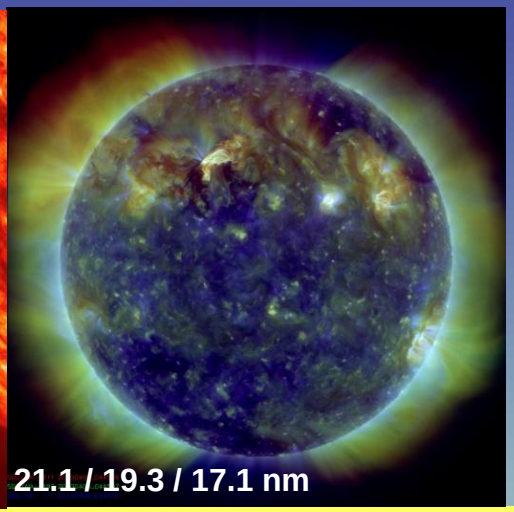
Multilayers with high performance and long lifetime are needed for EUV solar physics and space weather missions



7 EUV wavelengths,
9.4 nm to 33.5 nm.
Mo/Y, Mo/Si, SiC/Si.

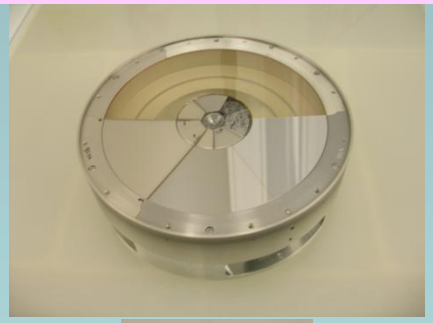


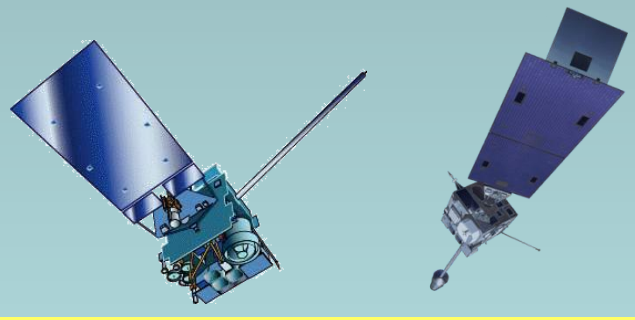




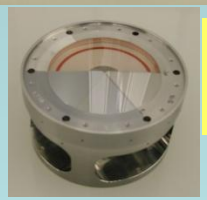
R. Soufli, *et al*, Proc. SPIE 5901, 59010M (2005).
R. Soufli, *et al*, Appl. Opt. 46, 3156-3163 (2007).
P. Boerner *et al*, Solar Physics (2011).
J. R. Lemen *et al*, Solar Physics (2011).

NASA's Solar Dynamics Observatory (SDO). Launch date: February 11, 2010.
http://sdo.gsfc.nasa.gov









Multilayer-coated test mirrors for NASA/NOAA's GOES-R space weather satellite. 6 EUV wavelengths, 9.4 nm to 30.4 nm (Mo/Y, Mo/Si).
Launch date: 2014

D. Martinez-Galarce, *et al*, Proc. SPIE 7732, 7732-177 (2010).

Reflective X-ray Optics LLC

LOCKHEED MARTIN

NASA

NOAA

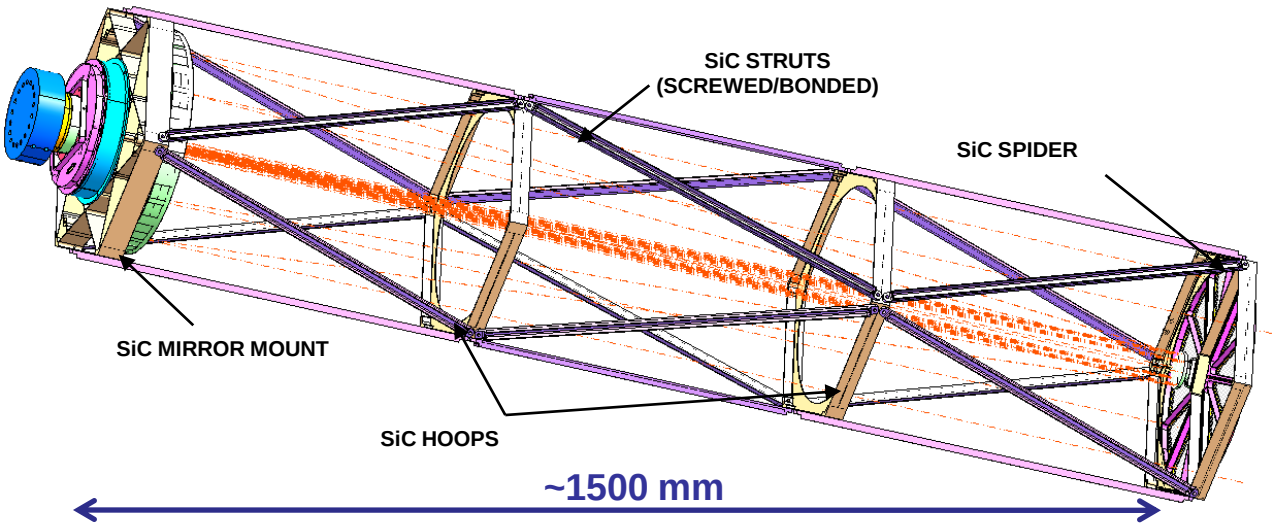
GOES R

SOLAR DYNAMICS OBSERVATORY

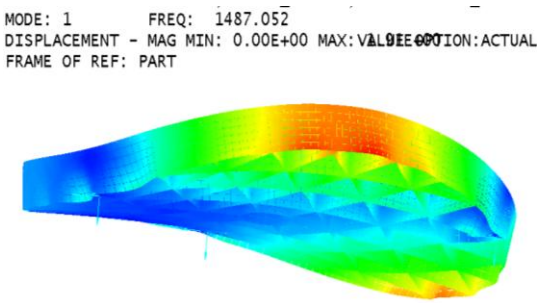
Atmospheric Imaging Assembly AIA

BERKELEY LAB

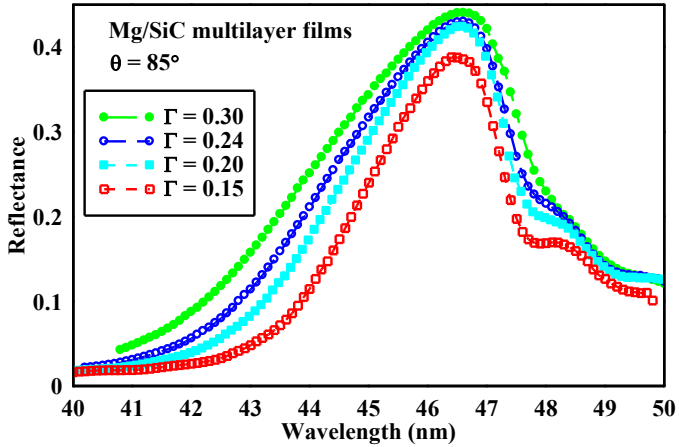
HiLiTE solar mission is designed to operate at 46.5 nm



HiLiTE: a 300-mm aperture Cassegrain telescope, aiming to study the Sun's transition region at the 46.5 nm Ne VII emission line. Designed to be made entirely of SiC, including optical substrates and metering structure.



	System
Effective Focal Length	10.313 m
Focal Ratio	f/34
Plate Scale	20 arcsec/mm
Field of View	> 4x4 arcmin*
	Primary Mirror
Clear Aperture	300 mm
Radius of Curvature	- 3564.2564 mm
Conic	- 1
	Secondary Mirror
Clear Aperture	40 mm
Radius of Curvature	- 680.89795 mm
Conic	- 2.0116



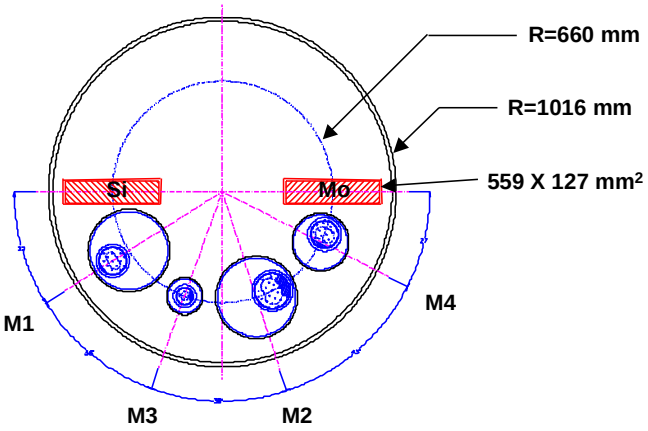
D. S. Martínez-Galarce, P. Boerner, R. Soufli, B. De Pontieu, N. Katz, A. Title, E. M. Gullikson, J. C. Robinson, S. L. Baker, "The high-resolution lightweight telescope for the EUV (HiLiTE)", Proc. SPIE 7011 , 70113K (2008).

Regina Soufli
regina.soufli@llnl.gov

LLNL facilities used for multilayer deposition and characterization of SiC/Mg multilayers discussed in this presentation

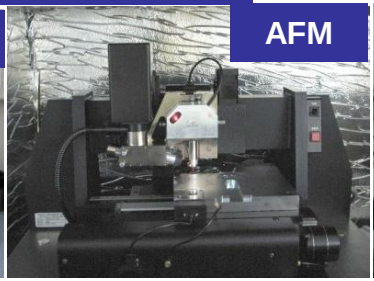
DC- magnetron sputtering multilayer deposition system





Precision surface metrology





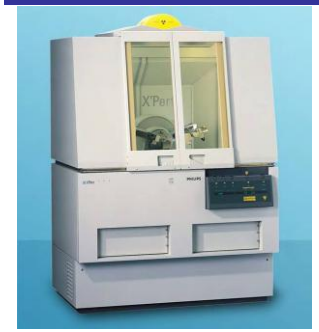


Also (not pictured):
- Contact profilometers
- Thin film stress measurement apparatus

Custom cleaning facility for optical substrates



X-Ray Diffractometer

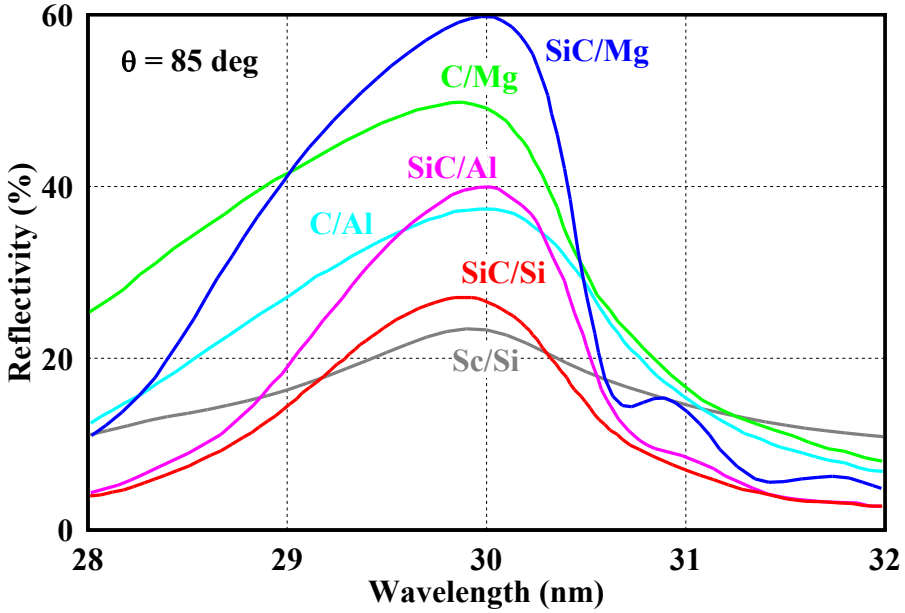


Regina Soufli
regina.soufli@llnl.gov

SiC/Mg could be the best-performing multilayer in the 25-75 nm wavelength region, except …

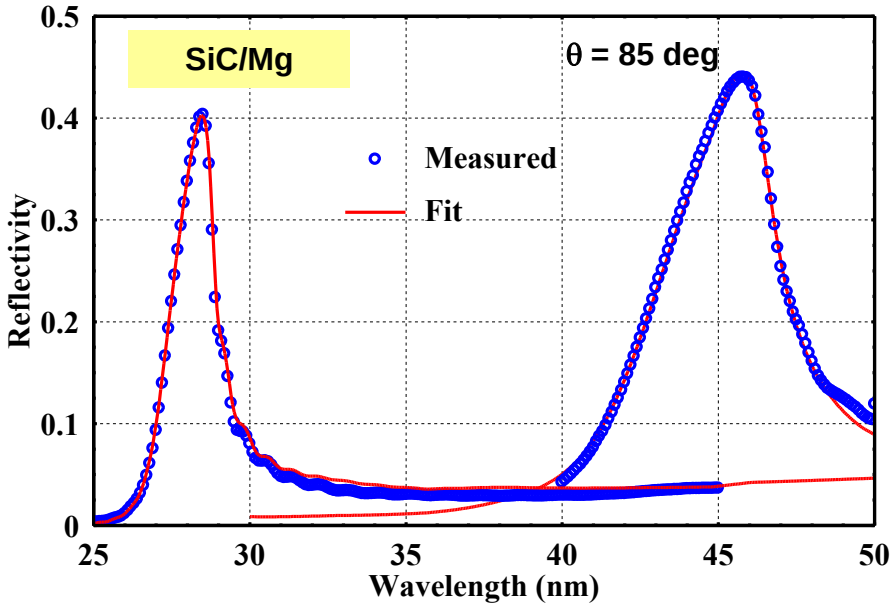
SiC/Mg combines high reflectivity, near-zero stress, high thermal stability (~ 300° C) and good spectral selectivity compared to other candidate multilayers

Calculated ideal reflectivity (roughness=0) for different multilayers



H. Takenaka *et al.*, J.of El. Spectr. and Rel. Phen. 144-147, 1047 (2005).

Experimental results and modeling of SiC/Mg multilayers deposited at LLNL



Measured at ALS beamline 6.3.2 (LBNL)
IMD modeling software by D. L. Windt

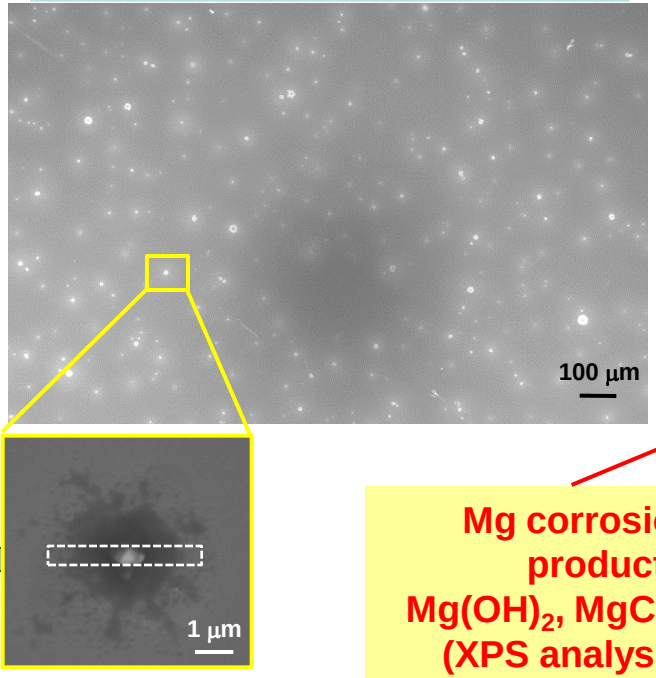
D. S. Martínez-Galarce, P. Boerner, R. Soufli, *et al,* Proc. SPIE 7011 , 70113K (2008).

Regina Soufli
regina.soufli@llnl.gov

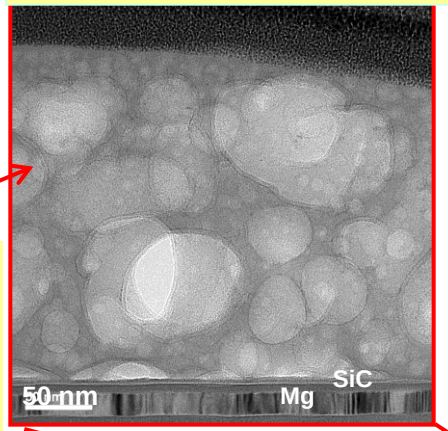
…corrosion prevents SiC/Mg use in applications that require stable performance and long lifetime

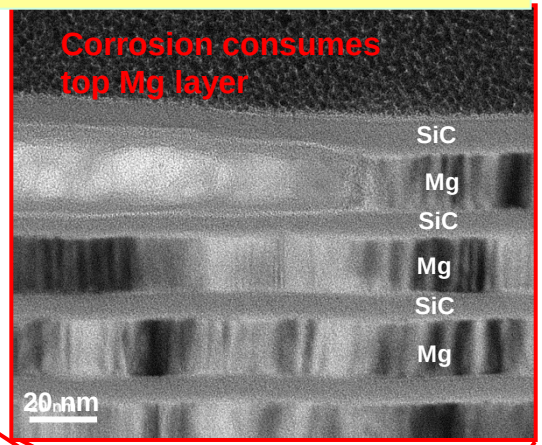
- ✓ Mg is the primary corrosive material
- ✓ SiC/Mg corrosion spots develop sporadically and expand over time
- ✓ Corrosion appears to start mostly from the top (unless multilayer is cleaved)
- ✓ Caused by humidity and contamination propagating and "finding" Mg through nano-voids in sputtered film

Top view (SEM) of corrosion spots on SiC/Mg after 3 years of aging

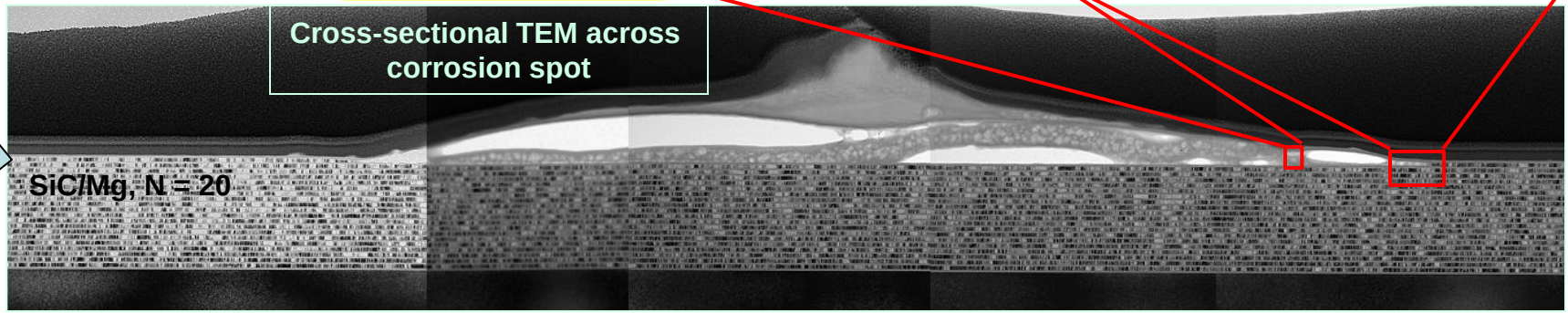


Mg corrosion products: $Mg(OH)_2$, $MgCO_3$ (XPS analysis)





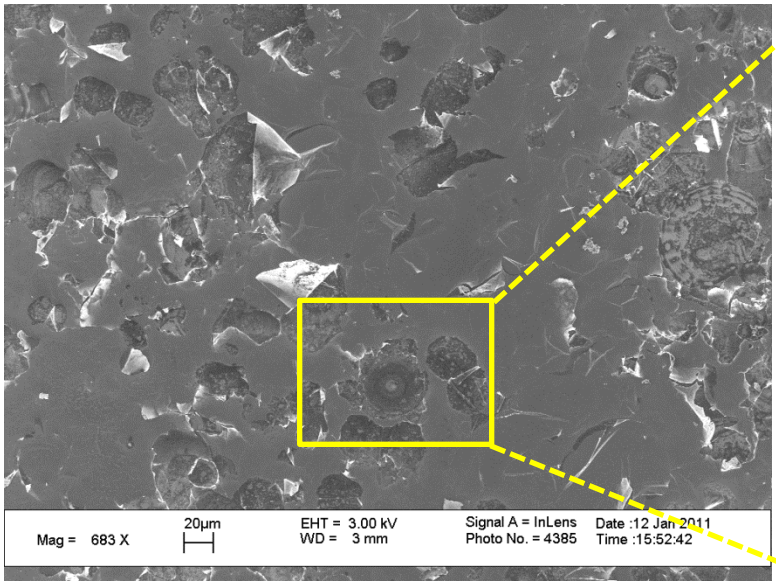
Cross-sectional TEM across corrosion spot

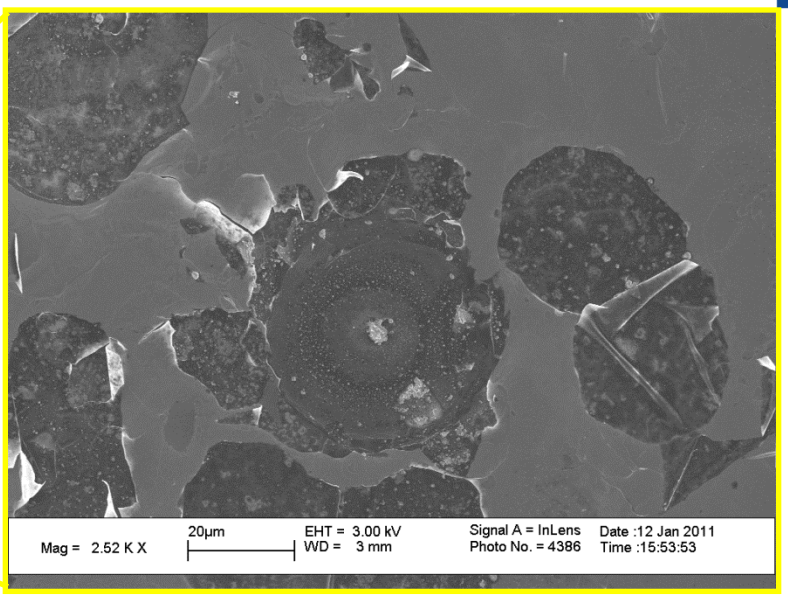


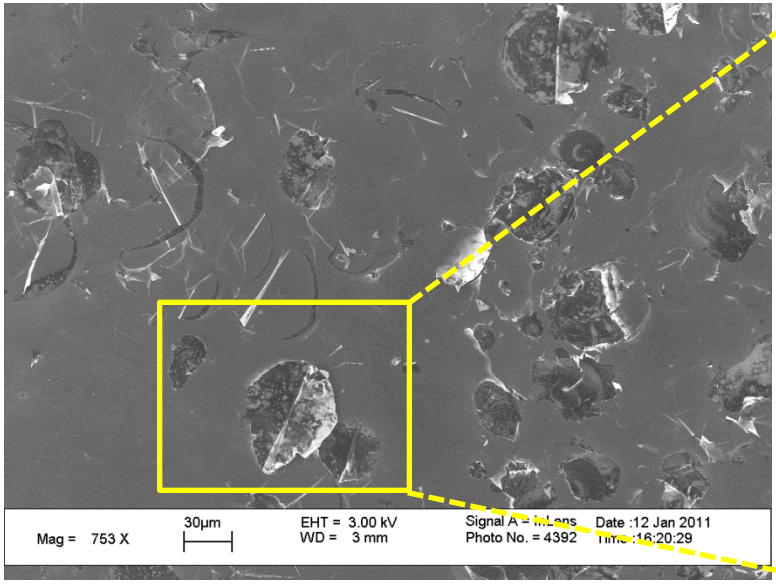
TEM measurements performed at EAG Labs, Sunnyvale (CA)

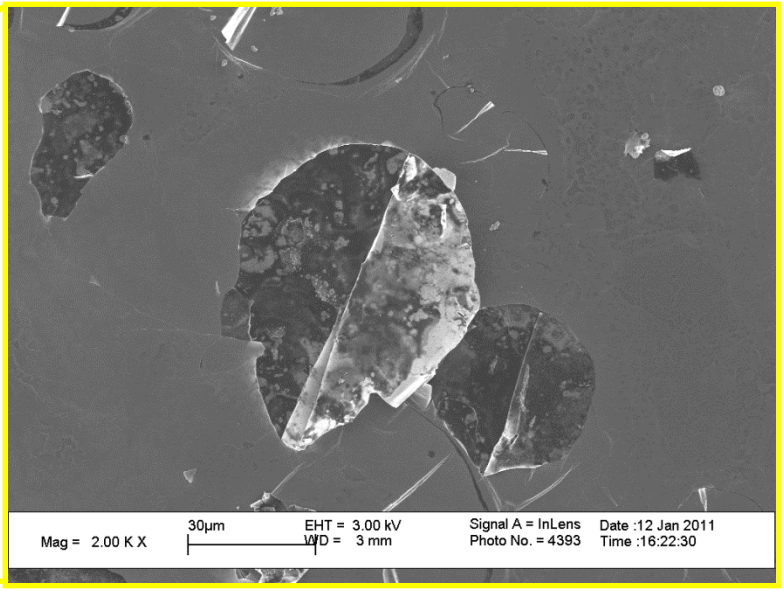
Regina Soufli
regina.soufli@llnl.gov

Top-view SEM images of SiC/Mg areas with advanced stages of corrosion after aging for 2.7 years



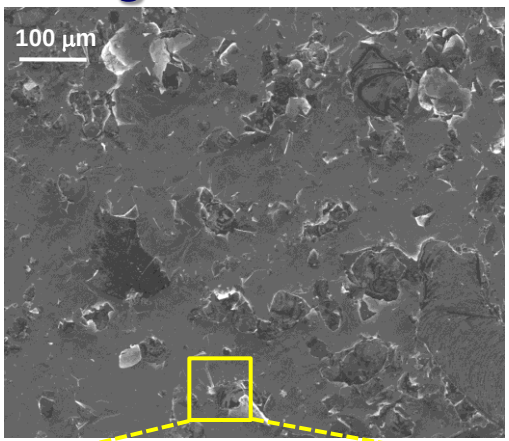


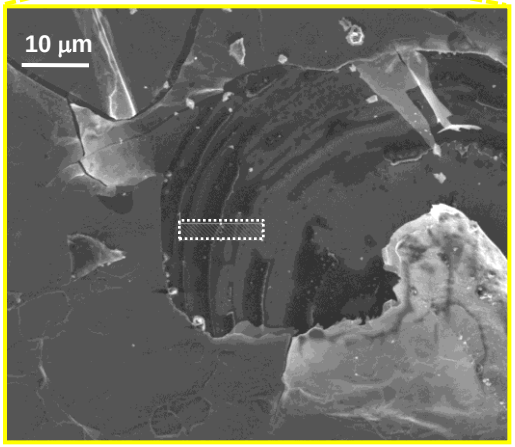




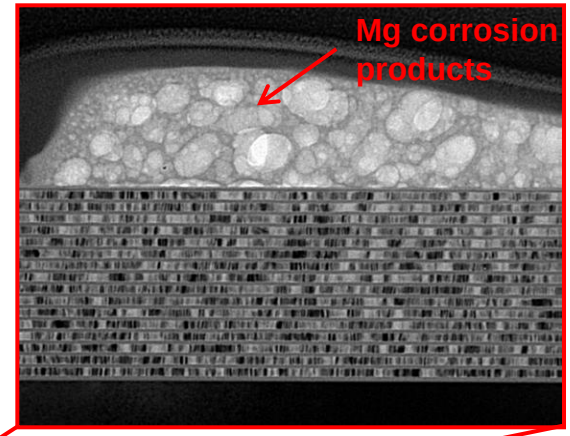
Regina Soufli
regina.soufli@llnl.gov

SEM and TEM images of SiC/Mg regions with advanced stages of corrosion after aging for 3 years

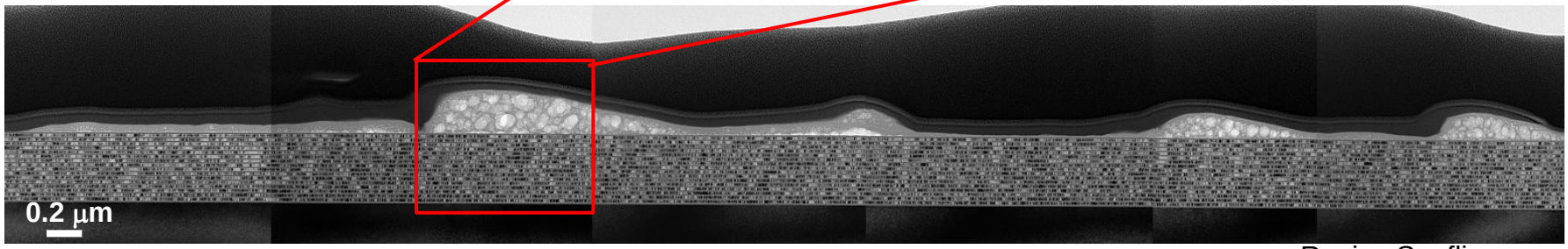




- Corrosion has prevented the use of SiC/Mg as EUV mirror coating in two recent NASA/NOAA solar physics missions: SDO and GOES-R. "Substitute" multilayers have much higher stress and reduced instrument throughput by 10X
- Future solar missions (Solar Orbiter, HiLiTE) and other applications are also affected



SiC/Mg, N = 16 (was N=20)
4 SiC/Mg bilayers are missing from top due to corrosion!



Regina Soufli
regina.soufli@llnl.gov

We have developed barrier layer structures to prevent SiC/Mg corrosion and extend lifetime, while maintaining high performance

✓ **Corrosion mitigation techniques developed for Mg in other industries (automotive, aerospace) are not suitable for high-performance, nanometer -scale EUV multilayers**

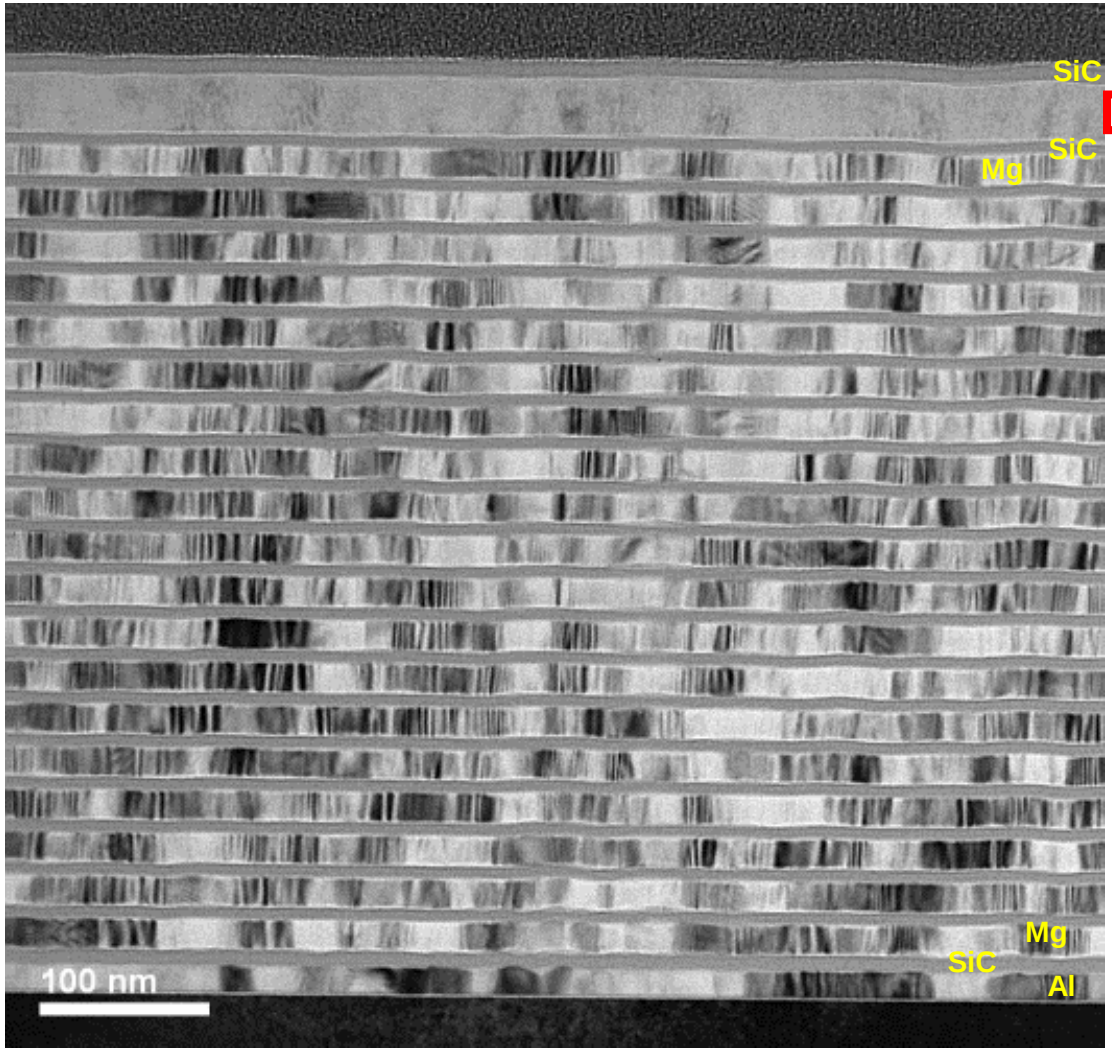
✓ **We have developed corrosion barrier structures for SiC/Mg multilayers**

✓ **Barrier layers are inserted at specific, key locations within the multilayer structure (not in each layer) to efficiently prevent corrosion while maintaining high reflectance and low stress**

Regina Soufli
regina.soufli@llnl.gov

Al-Mg intermixed layer underneath SiC capping layer acts as barrier against corrosion

SiC/Mg multilayer with Al-Mg corrosion barrier layer, aged for 2.5 years



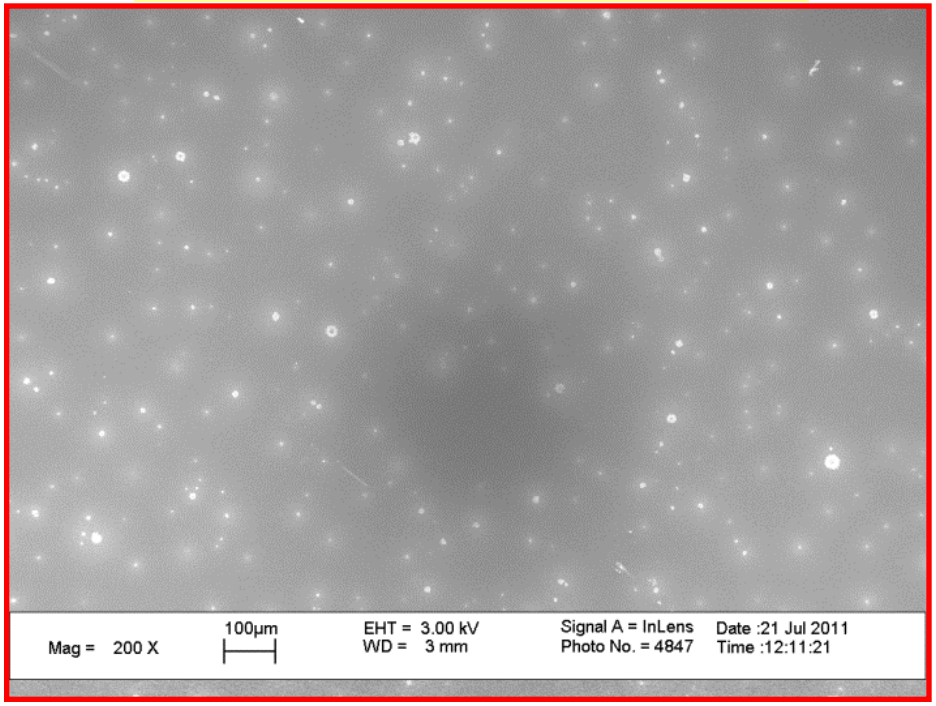
Al-Mg = Al (20 nm) + Mg (19 nm)

- ✓ **Mg and Al crystalline layers intermix completely to produce partially amorphous Mg-Al layer. Possible densification, less nano-voids**
- ✓ **Al is transparent in the 28-75 nm region, its thickness can be optimized to minimize impact in reflectivity**
- ✓ **XPS, EDX and LAXRD measurements indicate that the Mg-Al layer may be partially oxidized → additional protection against corrosion**
- ✓ **Mg-Al layer can be employed in additional locations within multilayer structure to protect against corrosion originating from multilayer or from substrate**

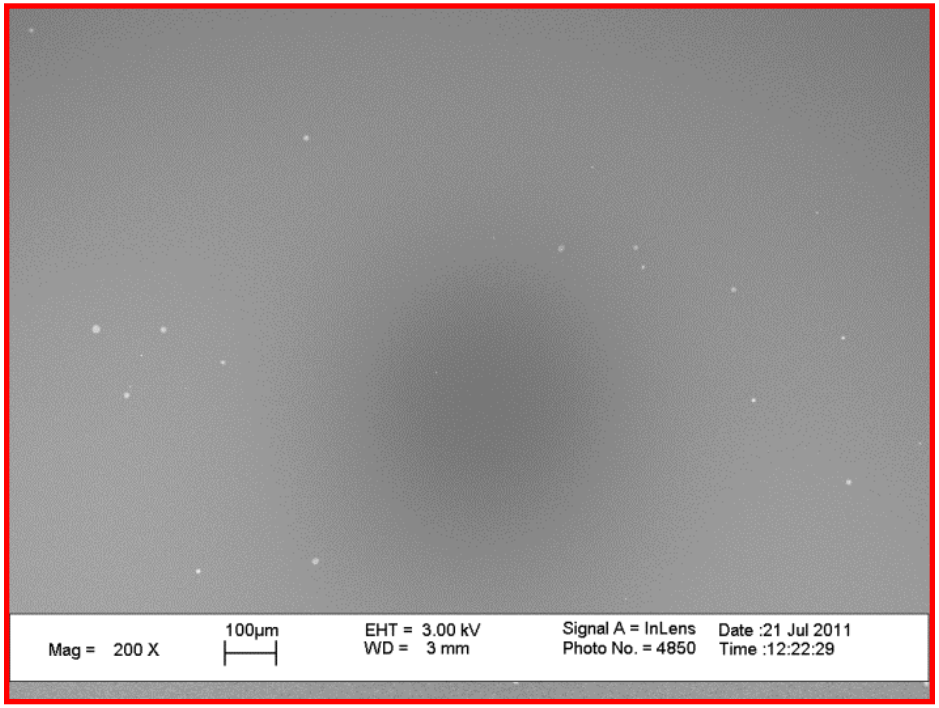
regina.soufli@llnl.gov

Corrosion barrier layers dramatically reduce SiC/Mg corrosion after aging for 3 years

Standard SiC/Mg (no barrier layers)

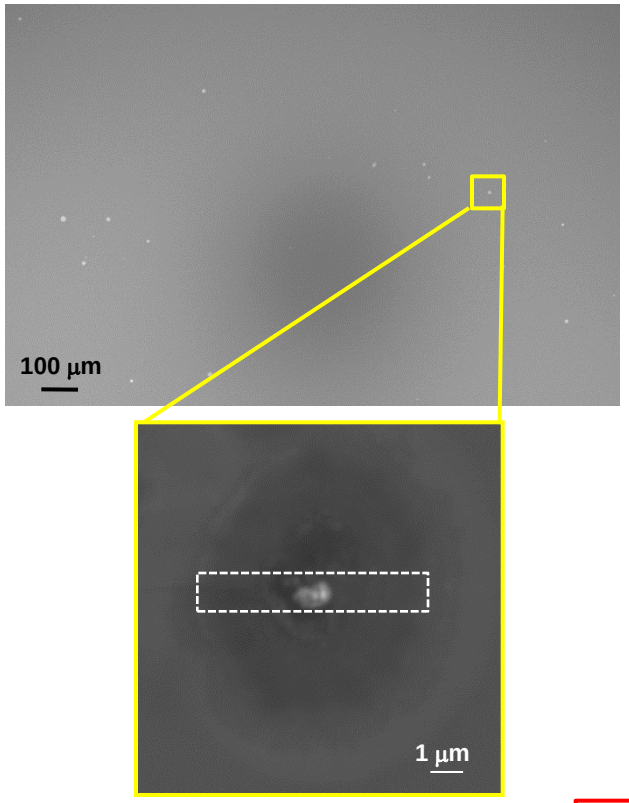


SiC/Mg with 2 barrier layers (top, bottom)

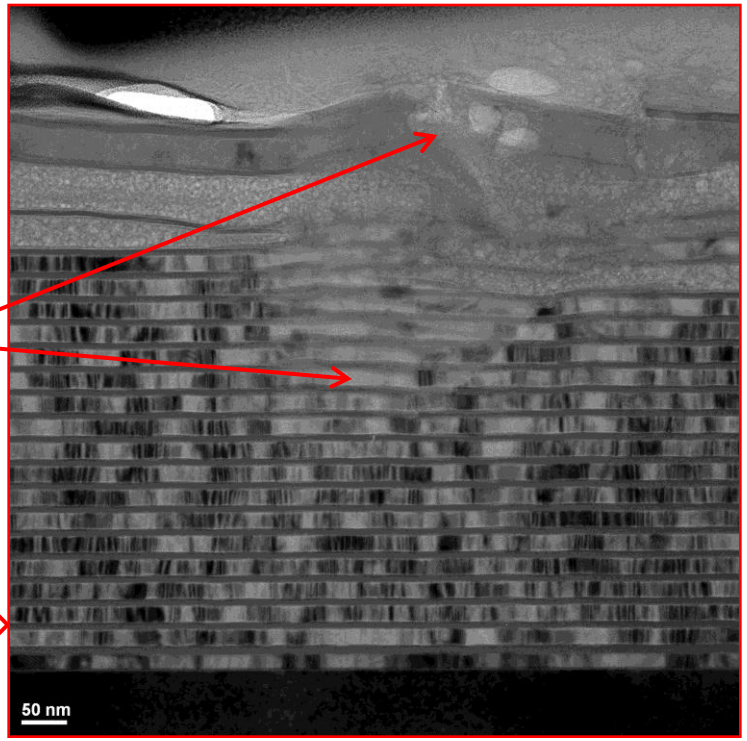


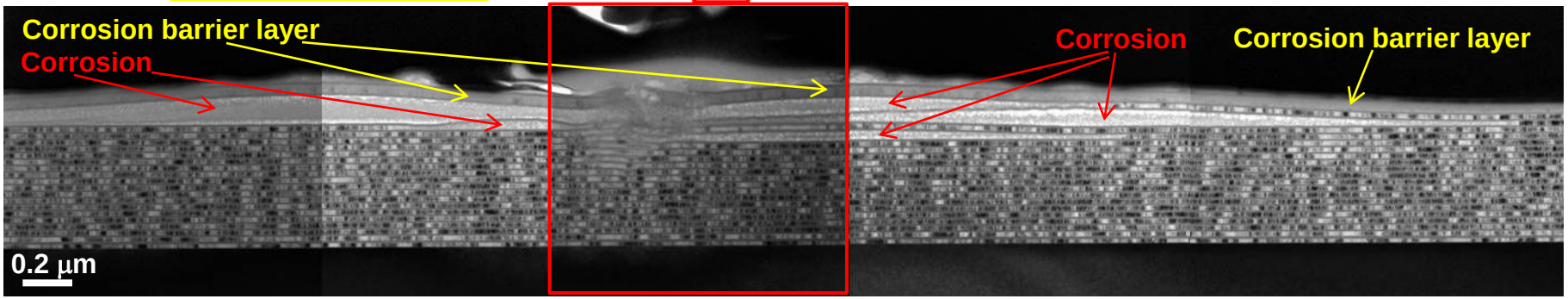
Regina Soufli
regina.soufli@llnl.gov

Study of corrosion spot on SiC/Mg with barrier layers after aging for 3 years



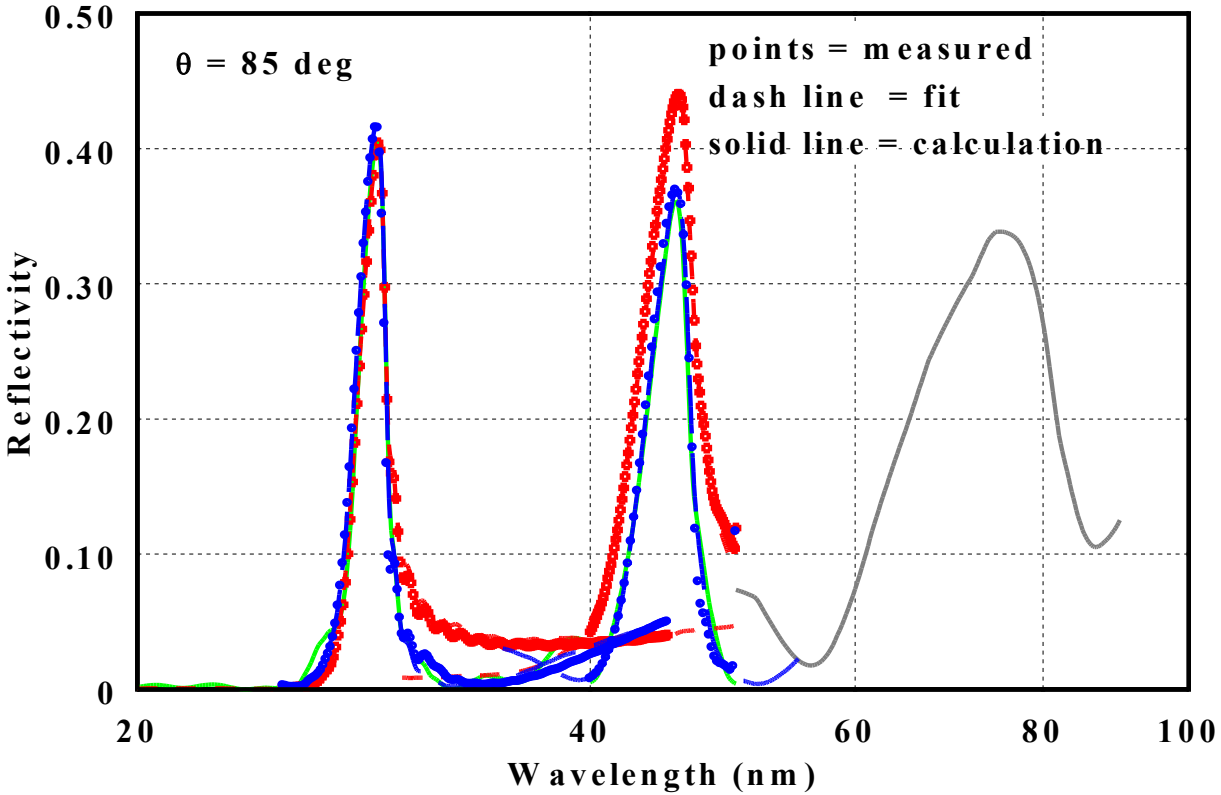
Possible corrosion origins





Regina Soufli
regina.soufli@llnl.gov

Experimental results and design concepts for SiC/Mg with corrosion barrier layers



Standard SiC/Mg (no barriers)

With 2 barrier layers (top, bottom)

With 3 barrier layers:

(top, bottom, inside multilayer)

N=2, with 2 barrier layers (top, bottom)

Measured at ALS beamline 6.3.2 (LBNL)
IMD modeling software by D. L. Windt

SiC/Mg with Al-based corrosion barrier layers may achieve very high reflectivity in the 75 nm wavelength region (based on calculations with available optical constants)

Regina Soufli
regina.soufli@llnl.gov

Summary and future work

- Corrosion has prevented the use of SiC/Mg multilayers in applications requiring good lifetime stability

- We have developed Al-based barrier layers that dramatically reduce corrosion, while preserving high reflectance and low stress

- The aforementioned advances may enable the implementation of corrosion-resistant, high-performance SiC/Mg coatings in the 25 – 75 nm region in applications such as tabletop EUV/soft x-ray laser sources and solar physics telescopes

- Further study and optimization of corrosion barrier structures and coating designs is underway

R. Soufli, M. Fernandez-Perea, J. C. Robinson, S. L. Baker, *et al*, "Corrosion-resistant , high- performance SiC/Mg multilayer interference coatings", in preparation.

Regina Soufli
regina.soufli@llnl.gov

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Regina Soufli
regina.soufli@llnl.gov